JACOBS

8/15/2019

Jenia McBrian
Quality Assurance Manager
Office of Air Quality Planning and Standards
Office of Air and Radiation
U.S. Environmental Protection Agency
109 T.W. Alexander Dr., Research Triangle Park, NC 27711

Subject: WA-3-176 / Mega PE and PM2.5 Round Robin Program Sampling, Gravimetric Analysis, and Distribution
Contract #: EP-C-15-008

Dear Ms McBrian:

Please find our response to the interrogatories as requested per your letter dated 8/2/2019 concerning the OAQPS Audit Findings of WA-3-176.

Sincerely,

Plessinger, Wendy

Digitally signed by Plessinger, Wendy
Date: 2019.08.15 17:03:51 -04'00'

Wendy Plessinger
Quality Assurance Officer
Jacobs Technology, Inc.

Enclosures

cc: Files: NRMRL Audit - data analysis – Jacobs.xlsx
Digby Email Sent 5-17-2019 - Follow-up materials to meeting on 5_14_2019.pdf
MTL AH225 PM Service by Scott Shultz 7-31-2019.pdf
MTL AH225 PM Service by Scott Shultz 7-31-2019.pdf
RiceLakeCertification_May2017 (300mg).pdf
RiceLakeCertification_May2017 (500mg).pdf
Weight Set 00386 2018-05-23 rpt (mg) (300mg).pdf

Weight Set 00386 2018-09-25 rpt (300 mg).pdf
Weight Set 00386 2019-06-04 rpt.pdf
Weight Set 00388 2018-05-23 rpt (mg) (500mg).pdf
Weight Set 00388 2018-09-25 rpt (500 mg).pdf
Weight Set 00388 2019-06-04 rpt.pdf

Interrogatories

1. What is the date of Jacobs most recent internal Quality Systems Audit and/or Management Systems Review?

   Jacobs Response: The most recent internal Quality Systems Audit / Management System Review was performed on-site by the Jacobs Technology corporate Quality Manager, Barbara Stevens, on 10/29/2018 - 10/31/2018.

2. How does Jacobs ensure that employees are complying with Jacobs Quality Management Plan?

   Jacobs Response: There are several ways in which Jacobs ensures compliance to the Jacobs Research Laboratory Support (RLS) Quality Management Plan.
   - *Annual Management System Review / Quality Systems Audits performed by the corporate Quality Manager.*
   - *Use of project-specific Quality Assurance Project Plans whether written by Jacobs or by EPA.*
   - *Use of Standard Operating Procedures which represent the EPA-approved method for performing research laboratory and field tasks. These are approved by EPA and by Jacobs.*
   - *Other types of audits such as technical systems, data quality and performance audits as prescribed in the project-specific QAPP or as requested.*
   - *New hire training performed for each new employee (Jacobs or teammate) which includes quality systems and laboratory notebook training.*
   - *Required annual review of the Jacobs RLS Quality Management Plan for all employees.*

3. Why were data from two separate weigh sessions, contained in separate data files, substituted for the original data collected on 11/17/2018 in Submissions 1 through 5?

   Jacobs Response: This was not a substitution of data but a use of post-shipment weights versus the use of pre-shipment weights in order to calculate final loaded weights. Data collected on 11/17/2018 consisted of pre-shipment loaded weights. Submissions 1 through 5 reflect the use of post-shipment loaded weights (collected 12/4/2018 through 12/7/2018) and pre-shipment tare weights (collected 10/23/2018) to calculate a final loaded weight. The Work Assignment Leader (WAL) was unsure as to which of the two tare weights and which of the two loaded weights should be used to calculate the final loaded weight: pre-shipment tare weights (weighed prior to shipping to secondary lab) or post-shipment tare weights (weighed after receipt from secondary lab) and pre-shipment loaded weights or post-shipment loaded weights.

   The post-shipment loaded weights used in these submissions consisted of weighings performed on 12/4/19 through 12/7/2019. It appears that the instrument may have stopped before completing all weighings in the first analysis on 12/4 to 12/5 (File: RR 2018 Raw data – pulled by OAQPS/RR Loaded Post1) so that the remaining weighings were run in a second analysis on 12/6 to 12/7 (File: RR 2018 Raw data – pulled by

OAQPS/RR Loaded Post2). Thus, post-shipment loaded weights collected in two different weighing runs were used in submissions 1 through 5.

In submissions 1 through 5, the WAL did not indicate which type of weight was being used to calculate final loaded weights. The WAL should have outlined in writing where post-shipment versus pre-shipment weights were being used, whether in the laboratory notebook, the data submission or via email.

Please also see the responses for questions 4 and 5 for additional details.

4. Why were original data collected on 11/17/18 manipulated to appear to be correct in the data summary in Submission 6?

Jacobs Response: Some of the filter IDs in the pre-shipment loaded raw data collected on 11/17/2018 were later found to be incorrect. During the pre-shipment weighing of the filters, some of the filters were inadvertently placed in the wrong filter carriers, and thus, the filters were mis-labeled in the MTL software. Unfortunately, this was not noticed at the time, and all the filters were shipped out to their respective labs on the same day they were weighed due to time constraints. The pre-shipment loaded weighing had to be done quickly so that the filters could be shipped out to be weighed before Thanksgiving 2018 (11/22/18). One of the round robin laboratories responsible for the secondary weighing of the filters was scheduled to be shutdown starting after Thanksgiving.

The first time that the mis-labeled filter error was noticed was when the particulate matter loadings were first calculated using the pre-shipment loaded weights in late March to early April of 2019. There were several filters for which the weights were off by milligrams instead of micrograms (as is normally the case). It was thought that the error had occurred during transfer of the filters to the filter carriers used with the MTL roboweigher and that some of the filter IDs, therefore, were incorrectly assigned in the software. In May 2019, after returning from a lengthy field study in Missoula, MT, the WAL attempted to cross-check the weights for the loaded pre-shipment filters with the post-shipment loaded weights in order to see if this had been the error made. It was ascertained that the filters were in the correct numerical order by filter ID but were recorded incorrectly in the software because the filters were placed in the wrong filter carriers. This is why the filter IDs were shifted in submission 6 (file: 20190517_KD_MasterResults_OAQPS-RR-2018-Fall.xlsm) and this is why the original raw data obtained from the MTL software by OAQPS (file: RR 2018 Raw data – pulled by OAQPS.xlsx) does not match the submission. This shifting of filter IDs is highlighted using brackets and arrows in the ORD\NRMRL QA audit which Jacobs has modified for clarification (file: NRMRL Audit – data analysis – Jacobs.xlsx). Please find this file included with this response.

The WAL thought that cross-checking the weights would be enough to justify re-assigning weights to the appropriate filter IDs. However, the WAL did not record this in the laboratory notebook, and the WAL did not communicate in writing with the WACOR

about this issue prior to the submissions being made. Additional time was lost between the 11/17/18 weighing session and the time of this discovery due to the January 2019 government shutdown during which Jacobs was ordered to stop work. Additionally, the WAL was sent off site for another work assignment to Missoula, MT on a 25-day field study from April 14 – May 12, 2019. In May 2019, the WAL communicated with the WACOR and OAQPS QA concerning these issues via email (5/17/2019; to: Jenia McBrian, Nealson Watkins and Ryan Stokes). In addition, the Jacobs QAO has been working with the WAL to ensure his communications are written, either via email or in the laboratory notebook. Kyle has made great improvement in this area.

Please see our response to question # 5 for further details.

5. Why were the original raw data logs from 11/17/18 (in Submissions 7 and 8) manipulated to appear to be correct when compared to the associated data summary in Submission 6?

   Jacobs Response: In order to reflect that the original raw data generated on 11/17/2018 contained incorrect filter ID labeling, the WAL adjusted a spreadsheet exported from the MTL computer which contained the raw data so that the shifted filter IDs would be evident. The original raw data as found within the MTL software were ***never*** *changed. The WAL sent an email dated 5/17/2019 to Jenia McBrian, Nealson Watkins and Ryan Stokes outlining the issues with the filter IDs and the submission of raw data reflecting corrected filter IDs:*

   "In going through and scrutinizing these data once more, I noticed that in the pre-shipment loaded weights spreadsheet, the Filter Weighing Software (FWS) incorrectly labeled some of the filter IDs (i.e., - FWS identified filters out of order, but filters were weighed in the correct order according to the filter carrier assignment document on the PC). This was further verified by cross checking the data in the pre-shipment loaded weights spreadsheet with the values in the post-shipment loaded weights spreadsheet and with the initial weight values in the two tare weight spreadsheets. After checking these references, the filter IDs were corrected in the master raw data file accordingly. I did not include the uncorrected pre-shipment loaded raw data as to avoid confusion, but I have it on file and can provide that if necessary. And I can provide more details on all of this if necessary, as well." – Kyle Digby, May 17, 2019 @ 2:48 PM

   Please find a copy of this email included with this response.

6. Why was EPA not informed when QC criteria were not met?

   Jacobs Response: The WAL believed that the most critical QC criteria was that the blank filters (pre vs post weighings) were within 15 µg of each other and that the triplicate weighings of each filter were within 15 µg of each other. In fact, the WAL re-weighed filters that were not within ***5*** *µg of each other which is well within the 15µg limit.*

   The guidance document EPA method 2.12 mentions reweighing the working standard

which implies immediate reweighing, but this method is not written for the use of an automatic weighing instrument. However, a re-weigh of the working standards was done every 10 filters during a batch. This did not always result in a working standard reweigh which passed the +/- 3 µg criteria, but the WAL was under the impression that this criterion was not as critical as the 15 µg criteria and since the working standards were only occasionally outside of the +/- 3 µg criteria (considering the working standard re-weigh occurring every 10 filters) rather than consistently, it was not something that the WAL felt required further consideration. This was an inadvertent error on the part of the WAL which can be attributed to his misunderstanding of the requirements and insufficient communication with QA and the WACOR.

Additionally, the repeatability of the balance was not taken into account at this time because the automated system had a long history of having this issue, and it was assumed by the WAL that the repeatability was already looked into by EPA (NERL, OAQPS).

According to the guidance document EPA Method 2.12 section 10.6 #8):

"If the verified and measured values of a working standard disagree by more than 3 µg (i.e., three times the microbalance's repeatability), reweigh the working standard. If the two values still disagree, troubleshoot and take appropriate corrective action, which may include (1) reverifying the working standards against the laboratory primary standards and/or (2) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance."

7. Why was EPA not notified that there were ongoing issues with the balance?

Jacobs Response: Please see our response to question # 6. Additionally, the maintenance and care of the ORD NERL robotic MTL weighing system is not within the scope of this work assignment, however since the problem with balance repeatability was identified during the ORD/NRMRL audit, the WAL has worked diligently to resolve the issue. The consensus between NRMRL QA, Jacobs QA and the WAL was that the balance repeatability was most likely being affected by the Faraday cage and associated hand-welded pan in use at the time. The WAL pursued this among other ideas with the roboweigher manufacturer, MTL. On 7/31/2019, an MTL technician, Scott Shultz, came on site to service the roboweigher and associated microbalance. He replaced the Faraday cage/hand-welded pan with a newly machine fabricated custom pan which better accommodates the roboweigher auto filter placement function with the balance, and he replaced the slip clutch.

Please see service documentation and associated emails written by Kyle Digby included with this response.

8. Why were check weight calibration data not updated in the DAS?

Jacobs Response: The working standard certificates that the metrology lab generated in September 2018 were meant to be a verification of the working standards, not a

certification. As such, the previous certifications were still in place and not expired (performed by the AEMD Metrology Lab in May 2018), and therefore the weights were not updated. The WAL confirmed that the verified numbers from the metrology lab were within +/-2 µg of the certified weights as per guidance document EPA Method 2.12 Section 9.7.2 #8): "Subsequent measurements of Cw must be within +/-2 µg of the initial Cw value."

Please see weight certifications and verifications for the 300 mg and 500 mg weights associated with the data in question (from May 2018 and September 2018). For reference the weight certifications performed by Rice Lake in May 2017 and the most recent weight certifications performed by the Metrology lab in June 2019 have also been included.

9. Why did Jacobs provide no QA oversight on this WA?

   Jacobs Response: During the early part of 2018, Jacobs experienced the loss of two different Quality Assurance Officers, one to retirement and the other to another opportunity. One of the department managers stepped up to take on the roll as interim QAO until an appropriate candidate could be found and hired. A new QAO was hired in late November 2018. Wendy Plessinger, the current QAO, has a significant background in quality assurance from the pharmaceutical and commercial environmental testing industries as well as several years' experience working in a research environment. Since her hire date, she has been getting up to speed on all projects. Unfortunately, this process takes time with over 60 work assignments to attend to. Soon after she arrived to start work, the January government shutdown occurred which further delayed the process. Since June, she has been working with the WAL to improve the quality of data generated using the roboweigher.

10. Why were QC results not verified to be in specification after each weigh session?

    Jacobs Response: The WAL did verify the QC results as soon after each weigh session as possible. However, the WAL should have recorded his findings in a timelier manner.

    Please see our response to question 6 for further details.

11. Why were filter sample results not reviewed after each weighing session?

    Jacobs Response: The WAL did review the results for the filter samples as soon as possible after each weighing session. However, the WAL should have recorded his findings in a timelier manner.

    Please see our response to question 4 for further details.

12. Why was the laboratory notebook not maintained?

    Jacobs Response: This was an inadvertent mistake due to time constraints. The pre-

shipment loaded filter weights were taken quickly in order to send the filters out for secondary weighting to be performed before Thanksgiving. A lot of the data in question was recorded just prior to the January government shutdown, but the WAL had several things to take care of prior to stopping work, and as a result the notebook was left incomplete. After the shutdown, the WAL was also responsible for several things that had to be accomplished in order to start work again. The WAL inadvertently did not maintain the laboratory notebook. However, the Jacobs QAO has been working directly with the WAL since June 2019 to resolve this issue for future work.

13. What training has Jacobs provided Kyle Digby in gravimetric analysis, maintaining laboratory notebooks, QC methods, and scientific integrity?

    Jacobs Response: Each employee working under the RLS contract receives laboratory notebook documentation training, Jacobs RLS quality system training and Jacobs Code of Conduct training. As for gravimetric analysis, Kyle Digby was trained on weighing Teflon filters manually using EPA method 5 in December 2016 by Jerry Faircloth (Jacobs) under the Cookstoves Emissions work assignment (WA-1-006). As a graduate student at Georgia Institute of Technology, Kyle was responsible for collecting and analyzing filters for elemental and organic carbon using thermal optical transmittance, and analyzing filters for ammonium, sulfate and other ions using ion chromatography.

    Most recently, Kyle Digby attended the Mettler Toledo Good Weighing Practices (GWP) training held on 8/1/2019 and sponsored by Scott Moore (NRMRL/AEMD). Please see a copy of Kyle's Mettler Toledo GWP certificate included with this response.

14. What is Kyle Digby's experience in gravimetric analysis outside of this WA?

    Jacobs Response: Please see our response to question 13.

15. What is Jacobs procedure for assessing competency of personnel assigned to perform tasks under this contract?

    Jacobs Response: The Jacobs RLS Quality Management Plan outlines the following: "For routine protocols, the trainee will study written SOPs, observe an experienced employee performing the tasks, and then perform the work under the direct supervision of an experienced person. After performing the tasks to the satisfaction of the WAL, the trainee will be allowed to perform the task independently." However, this type of training is performed on an as needed basis. Given Kyle's previous experience with filter collection and analysis and with using complex instrumentation, Jacobs allocated this work assignment to him as the most proficient member on our team.

In conclusion, Jacobs agrees with the corrective actions, where currently applicable, as laid out in the ORD/NRMRL QA audit and we will continue to implement these actions and any further actions we deem necessary in order to ensure the quality of the data generated for WA-4-176

going forward. It should be noted that the replacement of the Faraday cage/pan with the newly fabricated MTL pan in the balance associated with the MTL roboweigher has tremendously improved the repeatability of the balance and was the main reason for the QC failures. This was outlined in two emails and associated spreadsheets from Kyle Digby sent on 7/31/2019, 8/1/2019 and 8/7/2019. These emails and spreadsheets are included with this response.